

学校编码: 10384

分类号_____密级_____

学 号: 20620080150147

UDC_____

厦 门 大 学

博 士 学 位 论 文

难处理铜金精矿常压氧化预处理
过程的工艺与动力学研究

Technological and Kinetic Study for Oxidation Pretreatment
Process of Copper-bearing Refractory Gold Concentrate
under Atmospheric Pressure

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论文提交日期: 2 0 1 1 年 8 月

论文答辩日期: 2 0 1 1 年 8 月

学位授予日期: 2 0 1 1 年 月

答辩委员会主席: _____

评 阅 人: _____

2011 年 08 月

XIAMEN UNIVERSITY

**Technological and Kinetic Study for Oxidation Pretreatment
Process of Copper-bearing Refractory Gold Concentrate
under Atmospheric Pressure**

A Dissertation Submitted to the Graduate School
in Partial Fulfillment of the Requirements for the Degree
Doctor of Philosophy

Field of **Industrial Catalysis**

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August 2011

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摘 要

随着高品位、易选冶金矿资源的日益减少，如何开发利用复杂难处理金矿资源已成为亟需解决的重要课题。因传统火法冶金工艺的高能耗与高污染等问题日益凸显，湿法冶金技术成为了当前研究与应用的热点。对于难处理含铜金矿，湿法冶金的关键是将影响提金的含铜矿物分解浸出（称之为氧化预处理）。目前的研究绝大多数集中在热压浸出与生物浸出预处理方法上，而常压化学氧化预处理过程的研究相对较少。

本文中采用硫酸-氧介质于常压条件下对主要含黄铜矿与黄铁矿的铜金精矿进行氧化预处理，全面考察了铜金精矿氧化浸出工艺参数的影响并优化了工艺条件；系统研究了氧化浸出过程宏观动力学；深入分析了氧化浸出过程若干机理；初步探索了难处理金精矿中硫资源化的可能性。主要研究工作及结果如下：

1. 全面考察了硫酸-氧介质中铜金精矿常压氧化浸出过程工艺参数的影响，并完成了工艺条件优化。

(1) 考察了铜金精矿常压氧化浸出过程工艺参数的影响，获得了搅拌转速、氯化钠初始浓度、硫酸初始浓度、曝气气体组成、氧气流量、反应温度、矿浆浓度、反应时间与矿物粒度等工艺参数对铜金精矿中铜的浸出效果与铁的浸出及转化的影响规律。

(2) 通过正交设计实验重点研究了反应温度、反应时间、氧气流量、硫酸初始浓度与氯化钠初始浓度等5个关键因素的影响，获得了常压氧化浸出过程的最佳工艺条件：反应温度98 °C、反应时间12 h、氧气流量100 mL/min、硫酸初始浓度80 g/L、氯化钠初始浓度1.0 g/L，此工艺条件下铜浸出率可达89.7%。方差分析结果表明，在所考察水平范围内，反应温度的影响非常显著；硫酸初始浓度与反应时间依次次之，亦属于显著影响因素；氧气流量的影响显著性不高，而氯化钠初始浓度的影响最小，且不具显著性。

2. 系统研究了难处理铜金精矿在硫酸-氧介质中的常压氧化浸出过程动力学，建立了氧化浸出过程总体速率表达式。

(1) 为浸出过程动力学研究消除了外扩散影响并确定了适合的矿浆浓度条件。

铜金精矿常压氧化浸出体系存在的外扩散影响于250 rpm搅拌转速下基本可消除。较低矿浆浓度下，铜浸出速率受矿浆浓度影响显著；而在10 g/L矿浆浓度及以上，影响程度较小。

- (2) 研究并获得了铜金精矿在硫酸-氧介质中的常压氧化浸出过程的宏观动力学参数值，氧化浸出过程相对于硫酸浓度的表观反应级数为0.68；相对于氧气分压的表观反应级数为0.51；相对于氯化钠浓度的表观反应级数为0.30；相对于颗粒粒径的表观反应级数为-1.0。浸出反应的表观活化能因反应温度范围不同可分为：24.9 kJ/mol (22 ~ 70 °C) 与90.2 kJ/mol (70 ~ 98 °C)。建立了常压氧化浸出过程总体速率表达式。在70 ~ 98 °C温度范围内，铜金精矿常压氧化浸出过程宏观动力学的总体速率可表示为：

$$\frac{t}{\tau} = 1 - (1 - X)^{\frac{1}{3}} = 3.05 \times 10^9 \cdot \exp\left(-\frac{10850}{T}\right) \cdot C_H^{0.68} \cdot C_S^{0.30} \cdot P_O^{0.51} \cdot d_0^{-1.0} \cdot t。$$

3. 深入研究了难处理铜金精矿常压氧化浸出过程中溶解氧、铁离子与氯化钠的影响及其作用。

- (1) 铜金精矿常压氧化浸出体系中溶解氧对于氧化浸出反应至关重要。此外，氧化浸出过程中所溶出的铁以 Fe^{2+} 与 Fe^{3+} 的形式存在，其中 Fe^{3+} 在浸出过程中充当了浸出体系的另一个氧化剂。一方面， Fe^{3+} 在浸出过程中作为氧化剂大大促进了铜金精矿的氧化浸出；另一方面，浸出体系中溶解氧的存在保证了 Fe^{2+} 向 Fe^{3+} 的转化，使 Fe^{3+} 得以再生。

- (2) 氧化浸出过程所添加氯化物中的阳离子对浸出过程具一定的影响，采用碱金属与碱土金属的氯化物均有助于加强铜金精矿的氧化浸出，且前者效果更优。此外，氯化钠通过其中的钠离子与氯离子对浸出过程产生影响，钠离子主要是在促进铁离子水解形成黄钾铁矾类化合物上起作用；氯离子可能通过参与到黄铜矿氧化浸出过程中间产物的形成而起作用。

4. 初步探索了难处理铜金精矿中硫资源化的可能性。

- (1) 考察了浸出过程工艺参数对浸出渣中单质硫含量的影响，获得了氧化浸出工艺条件，包括氯化钠初始浓度、硫酸初始浓度、氧气流量、反应温度、矿物粒度与矿浆浓度等，对氧化浸出后浸出渣中单质硫含量的影响规律。

(2) 以浸出渣中的单质硫含量为指标, 采用均匀设计实验方法初步优化了氧化浸出过程的关键条件。在反应温度96 °C、反应时间13 h、氧气流量5 mL/min、硫酸初始浓度100 g/L, 氯化钠初始浓度20 g/L的条件下浸出渣中单质硫含量可高达14.6%, 即每吨浸出渣中可回收的硫磺可达到146 kg。此外, XRD、SEM与EDS表征结果表明, 所得产品为颗粒状的高纯度单质硫。

关键词: 难处理金矿; 黄铜矿; 常压氧化预处理; 工艺优化; 浸出过程动力学

Abstract

With dwindling reserves of high grade and easily workable gold mineral resources, recovery of gold from deposits with complex mineralogy has emerged as an important subject which needs to be solved urgently. Recently, out of energy and environmental consideration, focus is being shifted from the conventional roasting processes to the hydrometallurgical systems. With regard to the hydrometallurgy of copper-bearing refractory gold ores, the most important process is to decompose and leach the copper mineral matrices which have adverse effects on the gold extraction. So far, most of the work has been focused on the pressure leaching and bio-leaching processes, whereas the atmospheric leaching pretreatment process has been seldom explored.

The present work focused on the oxidation pretreatment of a copper-bearing refractory gold concentrate (RGC) with sulfide matrices mainly composed of chalcopyrite and pyrite. The extraction of copper from the RGC as well as the iron transform behaviors in sulfuric acid-oxygen media with the assistance of chloride under atmospheric pressure conditions was investigated. The influences of parameters on the pretreatment process were evaluated and the optimal conditions for the extraction of copper from the RGC were determined. Besides, the copper leaching kinetics of the pretreatment was studied systematically. Furthermore, special attention was also paid to the demonstration of the oxidation leaching mechanism. As well, a preliminary exploration concerning reclamation of elemental sulfur from the RGC was conducted. Main work and results are presented as follows:

1. The influences of parameters on the pretreatment process were evaluated and the optimal conditions for the extraction of copper from the RGC in sulfuric acid-oxygen media with the assistance of chloride under atmospheric pressure were determined, based on which the oxidation pretreatment process of the RGC has been

improved.

- (1) The influences of the pretreatment process parameters, including agitation speed, initial NaCl concentration, initial H₂SO₄ concentration, aeration gas composition, oxygen aeration rate, reaction temperature, pulp density, reaction time, and particle size, were systematically investigated and discussed. The results are of vital importance for enhancing the atmospheric oxidation leaching of the RGC.
- (2) Parametric optimization through orthogonal experimental design was conducted for the extraction of copper from the RGC. The optimal conditions were obtained as leaching temperature 98 °C, leaching time 12 h, oxygen aeration rate 100 mL min⁻¹, initial H₂SO₄ concentration 80 g L⁻¹, and initial NaCl concentration 1.0 g L⁻¹, yielding a copper extraction of 89.7%. Meanwhile, phase and surface characterizations of the leaching residues demonstrated that acid media containing dissolved oxygen and low content of chloride in general supported oxidation leaching of the RGC with high copper extraction. Furthermore, the experimental investigations demonstrated that three parameters, i.e. leaching temperature, initial H₂SO₄ concentration, and leaching time, were significant parameters in this leaching pretreatment system, whereas the other two were of less importance.

2. The copper leaching kinetics of the oxidation pretreatment process was studied, and the overall rate equation of the leaching process was established.

- (1) The effects of diffusion were eliminated through enhancing the agitation strength. The results showed that agitation speed had no significant effect on the initial copper leaching rate when maintained at 250 rpm or higher. Besides, the initial leaching rate would be significantly affected by the pulp density when which was below 10 g/L; nonetheless, when maintained at 10 g/L or higher, the influence of the pulp density on the initial leaching rate was not evident.
- (2) The leaching kinetics of the RGC was investigated in H₂SO₄-O₂ media under atmospheric pressure. It has been found that the leaching kinetic data conformed to the shrinking core model, and based on which the apparent reaction order with

respect to the H_2SO_4 concentration, O_2 partial pressure, NaCl concentration, and particle size were acquired as 0.68, 0.51, 0.30, -1.0 , respectively. The copper leaching kinetics were limited by the rate of surface reaction, with an apparent activation energy of 24.9 and 90.2 kJ/mol over the temperature range $22 \sim 70^\circ\text{C}$ and $70 \sim 98^\circ\text{C}$, respectively. Based on the above parametric values, the overall rate equation of the leaching process over the temperature range $70 \sim 98^\circ\text{C}$ was established below:

$$\frac{t}{\tau} = 1 - (1 - X)^{\frac{1}{3}} = 3.05 \times 10^9 \cdot \exp\left(-\frac{10850}{T}\right) \cdot C_{\text{H}}^{0.68} \cdot C_{\text{S}}^{0.30} \cdot P_{\text{O}}^{0.51} \cdot d_0^{-1.0} \cdot t^\circ$$

3. The influences of the dissolved O_2 , iron ions, and sodium chloride on the atmospheric oxidation leaching of the copper-bearing RGC as well as their role were studied.

- (1) The dissolved O_2 is essential for the oxidation leaching process of the RGC under atmospheric pressure. In addition, the dissolved iron presented as both Fe^{2+} and Fe^{3+} in the leaching solution. Moreover, the presence of the dissolved O_2 in the leaching system ensured the oxidation of Fe^{2+} to Fe^{3+} which served as another important oxidant to facilitate the oxidation leaching of the RGC greatly.
- (2) The cations of the chloride exhibited some effects on the copper extraction. Alkali and alkaline-earth metallic chloride could enhance the oxidation leaching of the RGC, and the former showed more significant effect. The presence of Na^+ in the leaching system resulted in the preferred formation of jarosite-type compounds, and Cl^- might be involved in the formation of intermediate compounds during the oxidation leaching of chalcopyrite.

4. A preliminary exploration concerning reclamation of elemental sulfur from the RGC was conducted.

- (1) The influences of the process parameters on the content of elemental sulfur in the leaching residues were investigated, including initial NaCl concentration, initial H_2SO_4 concentration, oxygen aeration rate, reaction temperature, pulp density,

and particle size.

- (2) Parametric optimization of the leaching process in terms of elemental sulfur reclamation through uniform experimental design was carried out. The optimal conditions were obtained as leaching temperature 96 °C, leaching time 13 h, oxygen aeration rate 5 mL min⁻¹, initial H₂SO₄ concentration 100 g L⁻¹, and initial NaCl concentration 20 g L⁻¹, yielding a sulfur content of 14.6% in the residue (i.e., an amount of 146 kg elemental sulfur could be acquired from per ton of leaching residue). And thus obtained sulfur was confirmed through phase and surface characterizations to be granular elemental sulfur with high purity.

Key words: refractory gold concentrate, chalcopyrite, atmospheric oxidation pretreatment, process optimization, leaching kinetics.

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第一章 绪论

1.1 引言

黄金，因其色泽而得名，是最早被人类发现并利用的金属之一，亦是最受欢迎的装饰品原料之一。金的密度大（ 19.3 g/cm^3 ）、熔点高（ $1063\text{ }^\circ\text{C}$ ），且具有极好的延展性、传热性和导电性；其化学性质稳定，在空气、碱及普通酸等环境中均能稳定存在，因此也决定了其在自然界中多以自然金的状态存在。然而，因其在地壳中含量稀少且分散而被归类为贵金属，该贵金属被赋予了商品和货币的双重属性，是世界各国货币信用体系的基础。除了作为货币与装饰品原材料之外，金因其独特的物理化学性质而在化工、电子等行业领域得到了广泛的应用。

冶金术的发明和应用在人类社会从蒙昧到文明的发展进程中起了重要作用，世界上各种古文明从史前时代到文明时代的转变通常是以金属采冶和加工技术的发明、金属工具的制作与使用为标志的^[1]。几千年前人类已掌握火法处理矿石以生产金属，而湿法冶金工艺则起步要迟得多，大体在炼金术士时代，酸和碱才开始被用于金属的冶炼^[2]。由于金的高稳定特性，其湿法冶炼直到王水的发现（Jabir Ibn Hayyan, 720 ~ 813 AD, 阿拉伯）才得以实现；而其真正实现跨越式发展是在十八世纪氰化提金工艺的应用之后^[2]。近几十年来，随着高品位、易开发金属矿产资源的不断减少，复杂难处理矿物受到越来越多的关注^[3]。冶金行业提到的难处理金矿（refractory gold ore），一般是指复杂、难冶的金矿石，这类矿石采用单一的常规氰化工艺难于达到有效提金的目的^[4-6]。因此，研究人员将大量精力集中在难处理金矿的预处理上，通常采用物理法、化学法或生物等方法将夹杂包裹金颗粒的矿体分解浸出，以使后续提金得以有效进行。

本章概述了金矿资源开发现状及冶炼技术的发展，重点综述了金属硫化物型含铜难处理金矿的预处理方法、过程原理、适用性与优缺点等，进而引出本研究的目的与内容。

1.2 金矿资源开发与黄金冶炼

1.2.1 黄金资源概述

据美国地质调查局《Mineral Commodity Summaries 2011》统计,截至 2010 年底,全球黄金储量为 51000 吨^[7]。按 2010 年全球黄金产量 2500 吨^[7]的开采水平计算,现有黄金资源储量静态保证年限约为 20.4 年。全球金矿资源储量主要集中在澳大利亚(约占世界总储量的 14.3%)、南非(11.8%)、俄罗斯(9.8%)、智利(6.7%)、美国(5.9%)和印度尼西亚(5.9%)等国家^[7]。金矿可形成于所有的地质时期和各种地质构造环境及岩石类型中,但分布不均衡^[8]。全球已发现的各种金矿床中,以脉金矿床、沙金矿床和多金属伴生金矿床为主^[9]。全球黄金开采中约 40%来自砂金,但近年来砂金在黄金产量中的比重有所下降,伴生金的比重则呈增大趋势;埋藏浅、品位高的金矿石正在大量减少^[8]。

中国黄金开采历史悠久,是世界上最早认识和开发利用黄金的国家之一^[10]。其金矿资源分布具有广泛但不平衡的特点,东部地区金矿分布广、类型多。从大区分布来看,东部地区黄金储量/资源量占 31.6%,中部地区黄金储量/资源量占 30.7%,西部地区黄金储量/资源量占 37.7%^[11]。中国黄金资源分布较广还体现在几乎各个省份均有探明的金矿资源;黄金保有储量/资源量在百吨以上的省(自治区)有 19 个。目前主要产金区是东部地区的辽宁、河北、山东、福建 4 省;中部地区的黑龙江、吉林、河南、湖北、湖南、江西、安徽 7 省;西部地区的内蒙古、新疆、甘肃、青海、陕西、四川、贵州、云南 8 省(自治区)^[12]。

总体上,中国的金矿资源具有如下特点:金矿资源分布广,但储量相对集中;矿床类型多,但以中小型矿床为主,大型矿床较少,可供露采的矿床很少;岩金矿为主,伴生金矿占有较大比例,砂金较少;金矿床中富矿少,中等品位金矿多,品位变化大^[9,11,13]。随着金矿的大规模开采,高品位、易选冶的金矿资源储量大大减少,赋存条件复杂、难采冶的金矿石数量增加^[14];金矿中微细粒、含硫、含砷、含碳等难处理金矿资源比重较大^[10,12]。中国东部地区许多大中型旧矿山的金矿资源已面临枯竭,大量黄金资源危机矿山出现。然而,深部找矿工作的成功表明中东部地区大多数老矿山的深部存在第二找矿空间;此外西部地区有巨大找矿前景。随着金矿选冶技术的发展,目前大量存放的低品位废石

和尾矿等将会成为可用资源^[13]；近年来，难处理金矿资源已逐渐得到开发^[10,12]。

1.2.2 金矿资源开发现状

随着黄金价格的持续上涨以及全球经济增长的减缓，黄金投资呈现明显上升趋势，全球黄金产量与消费量也持续上升。美国地质调查局《Mineral Commodity Summaries 2011》的统计数据显示，2010年中国黄金产量为345吨^[7]，居世界第一位。据世界黄金协会统计，2010年全球黄金需求量为3812.2吨，比上一年增长9%，近7年全球黄金需求量与黄金价格变化趋势如图1-1所示^[15]。同样由世界黄金协会提供的数据（图1-2）可看出，中国近几年来黄金需求量增长势头迅猛，现已是全球第二大黄金消费市场^[16]。

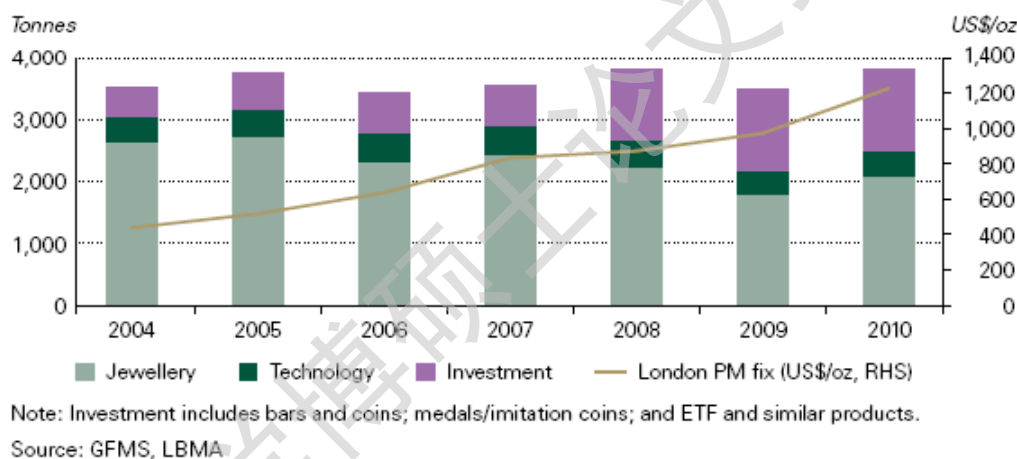


图1-1 全球黄金需求量与黄金价格

Fig. 1-1 Global gold demand and gold price^[15]

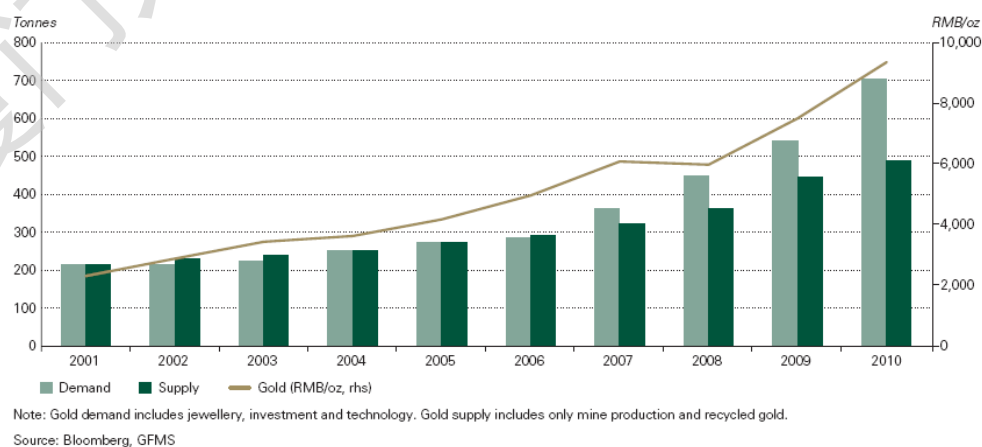


图1-2 中国黄金供需量与黄金价格

Fig. 1-2 China gold demand, supply and the gold price^[16]

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